National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2975

Diesel Particulate Matter (Industrial Forklift)

This Standard Reference Material (SRM) is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs) in diesel particulate matter and similar matrices. In addition to certified and reference values for selected polycyclic aromatic hydrocarbons, reference or information values are provided for total extractable mass, particle-size distribution, specific surface area, and the concentration of 1-nitropyrene. All of the chemical constituents for which certified, reference, and information values are provided in SRM 2975 are naturally present in the particulate material. A unit of SRM 2975 consists of a bottle containing 1 g of the diesel particulate matter collected from an industrial diesel-powered forklift.

Diesel particulate matter from the same lot of material that was used to prepare SRM 2975 was also used to prepare SRM 1975 Diesel Particulate Extract [1], which is a dichloromethane extract of the diesel particulate matter. A second diesel particulate material, SRM 1650a Diesel Particulate Matter [2], which was originally issued in 1985, is representative of heavy-duty diesel engine particulate emissions.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for 11 PAHs are provided in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values for the PAHs are based on the agreement of results obtained at NIST from two or more independent analytical methods.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 28 additional PAHs (some in combination) in Table 2. Reference values for total extractable mass and the particle-size distribution are provided in Tables 3 and 4, respectively. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. Explanations in support of each reference value are given as notes in Tables 2 through 4.

Information Concentration Values: Information values are provided for the concentration, expressed as mass fraction, of 1-nitropyrene in Table 5 and for specific surface area as determined by gas adsorption in Table 6. An information value is considered to be a value that may be of use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed.

Expiration of Certification: The certification of SRM 2975 is valid until **31 December 2009**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

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The coordination of the technical measurements leading to the certification of SRM 2975 was under the leadership of S.A. Wise of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by M.G. Vangel of the NIST Statistical Engineering Division.

The diesel particulate material was provided by M.E. Wright of the Donaldson Company, Inc., Minneapolis, MN.

Analytical measurements for the certification of SRM 2975 were performed at NIST by M. Lopez de Alda, D.L. Poster, L.C. Sander, M.M. Schantz, and L. Walton of the NIST Analytical Chemistry Division. Specific surface area, porosity, and 1-nitropyrene measurements were provided by P. Scheepers of the Department of Epidemiology at Katholieke Universiteit Nijmegen, Nijmegen, The Netherlands. The particle-size distribution data were provided by Honeywell, Inc., Clearwater, FL.

NOTICE AND WARNING TO USERS

Storage: SRM 2975 should be stored in its original bottle at temperatures less than 30 EC and away from direct sunlight.

Handling: This material is a naturally occurring diesel particulate material and contains constituents of known and unknown toxicities and mutagenicities. Therefore, appropriate caution and care should be exercised during its handling and use.

Instructions for Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed.

PREPARATION AND ANALYSIS[1]

Sample Collection and Preparation: The diesel particulate material used to prepare SRM 2975 was obtained from M.E. Wright of the Donaldson Company, Inc., Minneapolis, MN. The material was collected from a filtering system designed specifically for diesel-powered forklifts [3]. This diesel particulate material was selected based on a recommendation by J. Lewtas, US Environmental Protection Agency, Research Triangle Park, NC. The diesel particulate material was received at NIST in a 55-gal drum. The material was removed from the drum and homogenized in a V-blender for 1 h and then stored in polyethylene bags. A total of 13.7 kg of diesel particulate material was homogenized; a total of 5.65 kg of material was extracted for preparation of SRM 1975 and the remaining diesel particulate material was bottled for distribution as SRM 2975.

Polycyclic Aromatic Hydrocarbons (Tables 1 and 2): The general approach used for the value assignment of the PAHs in SRM 2975 was similar to that reported for the recent certification of several environmental matrix SRMs [4-7] and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or toluene/methanol mixture, cleanup of the extracts using solid phase extraction (SPE), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC) and (2) gas chromatography/mass spectrometry (GC/MS) analysis of the PAH fraction on three stationary phases of different selectivity, i.e., a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase, a 50 % phenyl-substituted methylpolysiloxane phase, and a smectic liquid crystalline stationary phase.

Seven sets of GC/MS results, designated as GC/MS (Ia and Ib), GC/MS (II), GC/MS (III), and GC/MS (IVa, IVb, and IVc), were obtained using three columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate subsamples of 100 mg from eight bottles of SRM 2975 were extracted with toluene:methanol (1:1 by volume) using PFE (excess volume in the PFE cells was filled with clean sodium sulfate) as described by Schantz et al. [8]. The extracts were concentrated to about 0.5 mL and placed on an aminopropylsilane SPE cartridge and eluted with 20 mL of 2 % DCM in hexane. The eluant was concentrated and then analyzed by GC/MS using a 0.25 mm i.d. x 60 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA) [GC/MS (Ia)]. A subset of 8 of the 16 extracts from GC/MS (Ia) were also analyzed on a 50 % (mole fraction) phenyl-substituted

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methylpolysiloxane stationary phase (0.25 mm i.d. x 60 m, 0.25 μm film thickness) (DB-17MS, J&W Scientific, Folsom, CA) [GC/MS (Ib)]. For the GC/MS (II) analyses, 100 mg samples of SRM 2975 were extracted with DCM using PFE; the extracts were processed and analyzed as described above for GC/MS (Ia). GC/MS (III) was identical to GC/MS (II) except that Soxhlet extraction with DCM for 18 h was used instead of PFE. For the GC/MS (IV) analyses, subsamples of 40 mg to 100 mg of SRM 2975 were extracted with DCM using PFE and the extracts processed as for GC/MS (I). The processed extracts were then analyzed by GC/MS using three different columns: 5 % phenyl methylpolysiloxane [GC/MS (IVa)], 50 % phenyl methylpolysiloxane [GC/MS (IVb)], and a 0.2 mm i.d. x 25 m (0.15 μm film thickness) smectic liquid crystalline phase (SB-Smectic, Dionex, Lee Scientific Division, Salt Lake City, UT) [GC/MS (IVc)].

For the LC-FL results, subsamples of approximately 200 mg from each of six bottles of SRM 2975 were Soxhlet extracted for 20 h using 200 mL of DCM. The extracts were concentrated and processed through aminopropylsilane SPE cartridges as described above for the GC/MS analyses. The processed extract was further fractionated using normal-phase LC on a semi-preparative aminopropylsilane column (μBondapak NH$_2$, 9 mm i.d. x 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions as described previously [9-11]. Four fractions were collected containing PAHs of molecular mass: 178 u and 202 u (fraction 1), 228 u (fraction 2), 252 u and 276 u (fraction 3) and 278 u (fraction 4). All of PAH fractions were analyzed using a 5-μm particle-size polymeric octadecylsilane (C$_{18}$) column (4.6 mm i.d. x 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [10-12]. For all of the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the diesel particulate samples prior to solvent extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PAHs: The homogeneity of SRM 2975 was assessed by analyzing duplicate samples of 100 mg each from eight bottles selected by stratified random sampling. Samples were processed and analyzed as described above for GC/MS (I). Statistically significant differences among bottles were observed for the PAHs at the 100 mg sample size, and this source of variability has been incorporated in the calculation of the uncertainty associated with the assigned values.

Nitro-Substituted PAHs (Table 5): The concentration of 1-nitropyrene was measured at the Department of Epidemiology at Katholieke Universiteit Nijmegen, Nijmegen, The Netherlands using the GC/MS method [13].

Mutagenicity: The mutagenicity of solvent extracts of SRM 2975 has not been determined; however, the mutagenicity values for SRM 1975 should be applicable to extracts of SRM 2975. The diesel particulate of SRM 2975 was used for the preparation of SRM 1975 Diesel Particulate Extract, a DCM extract of the SRM 2975 diesel particulate matter. SRM 1975 was prepared by Soxhlet extraction using DCM of multiple samples of SRM 2975. Reference values for mutagenic activity for SRM 1975 are reported in the Certificate of Analysis for SRM 1975 [1] and in detail in a publication [14].

Percent Extractable Mass (Table 3): For the determination of percent extractable mass, six subsamples of approximately 1 g to 2 g of SRM 2975 were extracted using Soxhlet extraction for 18 h with DCM. The extract was concentrated to approximately 20 mL and then filtered to remove particulate matter. Aliquots of 100 μL to 150 μL were placed in tared aluminum foil pans; the DCM was evaporated until constant mass was obtained and then the mass of the remaining residue was determined.

Particle-Size Information (Table 4): Dry particle-size distribution measurements for SRM 2975 were obtained as part of a collaborative effort with Honeywell, Inc., Clearwater, FL. A Microtrac® particle analyzer, which makes use of light-scattering techniques, was used to measure the particle-size distribution of SRM 2975. Briefly, a reference beam is used to penetrate a field of particles and the light that scatters in the forward direction from the field is measured and the particle size as a volume distribution is derived via a computer-assisted analysis. From these data, the total volume, average size, and a characteristic width of the particle-size distribution are calculated. The system has a working range from 0.7 μm to 700 μm.

Specific Surface Area and Porosity (Table 6): The specific surface area and porosity were determined based on nitrogen gas adsorption measurements [15]. The gas adsorption measurements were performed on a NOVA-1200 instrument, Quantachrome Corp., Boynton Beach, FL at 77 K after the samples were outgassed for 24 h at 120 EC under vacuum. The nitrogen isotherms were analyzed using the Brunauer-Emmet-Teller (BET) equation [16] to obtain the surface area (Table 6) and the Barrett-Joyner-Halenda (BJH) method [17] to obtain the porosity. Based on the BJH method, SRM 2975 shows a wide distribution of mesopores, but with substantial outer area. The pore diameter of the particles in SRM 2975 range from 4 nm to 35 nm with the greater number of particles at about 20 nm.

Table 1. Certified Concentrations for Selected PAHs in SRM 2975

	Mass Fractions (mg/kg)[a]		
Phenanthrene[b,c,d,e,f,g]	17.0	±	2.8
Fluoranthene[b,c,,d,e,f,g]	26.6	±	5.1
Pyrene[b,c,d,e,f,g]	0.90	±	0.24
Benz[a]anthracene[b,c,d,e,f,g,h]	0.317	±	0.066
Chrysene[g,h]	4.56	±	0.16
Triphenylene[g,h]	5.22	±	0.20
Benzo[j]fluoranthene[h,i]	0.82	±	0.11
Benzo[k]fluoranthene[c,d,e,f,g,h,i]	0.678	±	0.076
Benzo[e]pyrene[b,c,d,e,f,h]	1.11	±	0.10
Benzo[a]pyrene[e,f,g,h]	0.0522	±	0.0053
Benzo[ghi]perylene[b,c,e,f]	0.498	±	0.044

[a] Each set of results is expressed as the certified value ∀ the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods. For results from two methods, the certified value is the equally weighted mean; for results from three or more methods, the certified value is the mean weighted as described in Paule and Mandel [18]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [19], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method and among methods, as well as uncertainty due to the variation among the bottles. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %

[b] GC/MS (Ia) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with toluene:methanol mixture

[c] GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM

[d] GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM

[e] GC/MS (IVa) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM

[f] GC/MS (IVb) on 50 % phenyl-substituted methylpolysiloxane phase of same extracts as GC/MS (IVa)

[g] LC-FL of isomeric PAH fractions after Soxhlet extraction with DCM

[h] GC/MS (IVc) on a smectic liquid crystalline phase of same extracts as GC/MS (IVa)

[i] GC/MS (Ib) on 50 % phenyl-substituted methylpolysiloxane phase of selected extracts from GC/MS (Ia)

The concentrations for selected PAHs in Table 2 are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the agreement among results from multiple methods was insufficient for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Table 2. Reference Concentrations for Selected PAHs in SRM 2975

	Mass Fractions (mg/kg)[a]		
1-Methylphenanthrene[b,c,d,e,f]	0.89	±	0.11
2-Methylphenanthrene[b,c,d,e,f]	2.0	±	0.2
3-Methylphenanthrene[b,c,d,e,f]	1.0	±	0.2
4- and 9-Methylphenanthrene[b,c,d,e,f]	0.44	±	0.09
1,2-Dimethylphenanthrene[e,f]	0.05	±	0.02
1,6-, 1,7-, 2,5-, and 2,9-Dimethylphenanthrene[e,f]	0.57	±	0.08
1,8-Dimethylphenanthrene[e,f]	0.06	±	0.02
2,6-Dimethylphenanthrene[e,f]	0.25	±	0.05
2,7-Dimethylphenanthrene[e,f]	0.23	±	0.05
3,6-Dimethylphenanthrene[e,f]	0.18	±	0.02
Anthracene[e,g]	0.038	±	0.008
Benzo[ghi]fluoranthene[h]	10.2	±	0.5
8-Methylfluoranthene[f]	0.068	±	0.004
1-, 3-, and 7-Methylfluoranthene[e]	0.53	±	0.03
2-Methylpyrene[e,f]	0.040	±	0.008
4-Methylpyrene[e,f]	0.022	±	0.005
Benzo[c]phenathrene[b,c,d,e,f,i]	1.0	±	0.4
Benzo[a]fluoranthene[f,i]	0.06	±	0.02
Benzo[b]fluoranthene[f,h,i]	11.5	±	3.6
Perylene[g]	0.054	±	0.009
Indeno[1,2,3-cd]pyrene[e,f]	1.4	±	0.2
Indeno[1,2,3-cd]fluoranthene[f]	1.1	±	0.2
Dibenz[a,j]anthracene[e]	0.37	±	0.07
Dibenz[a,c]anthracene/Dibenz[a,h]anthracene[e]	0.52	±	0.08
Pentaphene[e]	0.038	±	0.007
Benzo[b]chrysene[e,f]	0.08	±	0.03
Picene[e,f]	1.0	±	0.2
Coronene[e]	1.1	±	0.2

[a] Each set of results is expressed as the reference value ∀ the expanded uncertainty. Each reference value is the mean from one analytical method or a mean of the means from two or more analytical methods. For results from two methods, the certified value is the equally weighted mean; for results from three or more methods, the certified value is the mean weighted as described in Paule and Mandel [18]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [19], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method and among methods, as well as uncertainty due to the variation among the bottles. The expanded uncertainty defines a range that contains the estimate of the true value at a level of confidence of approximately 95 %.

[b] GC/MS (Ia) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with toluene:methanol mixture

[c] GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM

[d] GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM

[e] GC/MS (IVa) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM

[f] GC/MS (IVb) on 50 % phenyl-substituted methylpolysiloxane phase of same extracts as GC/MS (IVa)

[g] LC-FL of isomeric PAH fractions after Soxhlet extraction with DCM

[h] GC/MS (IVc) on a smectic liquid crystalline phase of same extracts as GC/MS (IVa)

[i] GC/MS (Ib) on 50 % phenyl-substituted methylpolysiloxane phase of selected extracts from GC/MS (Ia)

The total extractable mass in Table 3 is provided as a reference value because the result is method specific as defined by the corresponding procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedure used, the reference value should be useful for comparison with results obtained using similar procedures.

Table 3. Reference Value for Total Extractable Mass for SRM 2975

Total Extractable Mass[a]	2.7 % ± 0.2 % Mass Fraction[b]

[a] Extractable mass as determined from Soxhlet extraction using DCM
[b] The results are expressed as the reference value ∀ the expanded uncertainty. The reference value for the total extractable mass is the mean value of six measurements. The uncertainty, computed according to the CIPM approach as described in the ISO Guide [19], is an expanded uncertainty at the 95 % level of confidence. The expanded uncertainty defines a range that contains the best estimate of the true value at a level of confidence of approximately 95 %.

Particle size results in Table 4 are provided as reference values because the results are method specific as defined by the corresponding procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedure used, the reference values should be useful for comparison with results obtained using similar procedures.

Table 4. Reference Values for Particle-Size Characteristics for SRM 2975

Particle Measurement	Value[a]		
Mean diameter (volume distribution, MV, μm)[b]	31.9	±	0.6
Mean diameter (area distribution, μm)[c]	11.2	±	0.1
Mean diameter (number distribution, μm)[d]	1.62	±	0.01
Surface Area (m^2/cm^3)[e]	0.538	±	0.006

The following data show the percent of the volume that is smaller than the indicated size:

Percentile	Particle Diameter (μm)[a]		
95	110	±	3
90	70	±	2
80	44.9	±	0.8
70	32.4	±	0.6
60	24.8	±	0.4
50[f]	19.4	±	0.3
40	15.2	±	0.2
30	11.7	±	0.2
20	8.5	±	0.1
10	5.3	±	0.1

[a] Each reference value is the mean value of measurements from the analysis of subsamples from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [19], is an expanded uncertainty at the 95 % level of confidence. The expanded uncertainty defines a range that contains the best estimate of the true value at a level of confidence of approximately approximately 95 %.
[b] The mean diameter of the volume distribution represents the center of gravity of the distribution and compensates for scattering efficiency and refractive index. This parameter is strongly influenced by coarse particles.
[c] The mean diameter of the area distribution, calculated from the volume distribution with less influence from the presence of coarse particles than the MV parameter.
[d] The mean diameter of the number distribution calculated from the volume distribution.
[e] Calculated specific surface area assuming solid, spherical particles. This is a computation and should not be interchanged with an adsorption method of surface area determination (see Table 6) as this value does not reflect porosity or topographical characteristics.
[f] Median diameter (50 % of the volume is less than 19.4 μm)

Table 5. Information Value for Concentration of Nitro-substituted PAHs in SRM 2975

Mass Fraction (mg/kg)

1-Nitropyrene	36

Table 6. Information Value for Specific Surface Area of SRM 2975 as Determined by Gas Adsorption

Specific Surface Area (S)[a]	91 m^2/g

[a] Specific surface area determined by multi-point N_2 adsorption BET method.

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Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: (30l) 975-6776, Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://www.nist.gov/srm.